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## MEMORANDUM

DATE: 11 January 1999

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle  
Rmm Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9806584  
Site Duwamish River

WORK ASSIGNMENT NO 46-35-0JZZ

WORK ORDER NO 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle (memo only)  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of nine sediment samples, laboratory batch K9806584, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98394020	98394021	98394022	98394027	98394028
98394029	98394030	98394033	98394034	

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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### 1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples were extracted 52 days after sample collection, exceeding the 7 day holding time criteria. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples.

### 2 Detection Limits

Detection limits met project required quantitation limits with the following exceptions:

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98394020	Tetrabutyltin	10	15
98394021	Tetrabutyltin	10	15
98394022	Tetrabutyltin	10	15
98394027	n-Butyltin	10	18
98394028	n-Butyltin	10	17
98394029	n-Butyltin	10	13
98394030	n-Butyltin	10	15
98394033	Tetrabutyltin	10	15
98394034	Tetrabutyltin	10	15

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference

### 3 Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

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#### 4 Continuing Calibrations

Continuing calibration check was performed after every 10 samples. Target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

#### 5. Blanks

##### a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

##### b) Field Blanks

No field blanks were associated with this SDG.

#### 6 Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, Triethyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	18 - 125%
Triethyltin	28 - 122%

Surrogate compound percent recoveries exceeded the QC limits for the following samples:

Sample	Surrogate	Percent Recovery
98394029	Tripropyltin	53



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Samples results and detection limits were qualified as estimated (UJ/J) when both surrogate recoveries were outside the QC limits

#### 7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 27 to 162 percent for Tetrabutyltin and Tributyltin, and 8 to 161 percent for Dibutyltin and n-Butyltin for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K981113-LCS	n-Butyltin	28	60-130 (P) 27-162 (L)	98394020 through 98394022 98394027 through 98394030 98394033 98394034

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were less than project limits

#### 8 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

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All MS/MSD sample percent recoveries and relative percent differences (RPDs) met QC guidelines.

9. Field Duplicate Analysis

Samples 98394027 and 98394029 were field duplicates. Analytes were detected in sample 98394027, but not detected in sample 98394029. The RPD value for Tetrabutyltin was less than 35 percent. Tributyltin results were qualified as estimated (J).

Samples 98394028 and 98394030 were also field duplicates. Analytes were detected in sample 98394030, but not detected in sample 98394028. Results were qualified as estimated (J).

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. A duplicate analysis was performed on sample 98394020; RPD values were less than 35 percent for analytes with concentrations greater than five times the reporting limit except Tributyltin, which had an RPD of 93 percent. This anomaly was noted in the case narrative. Tributyltin was qualified as estimated in sample 98394020. No other problems were noted in the case narrative.

11. Laboratory Contact

No laboratory contact was required

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

Data Qualifiers

U - The compound was analyzed for, but was not detected

UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met

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- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification).
- I - Elevated reporting limit due to matrix interference.

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**COLUMBIA ANALYTICAL SERVICES, INC.**

**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/22/98  
**Date Received:** 9/23/98

**Butyltins**

<b>Sample Name</b>	98394020	<b>Units</b>	ug/Kg (ppb)
<b>Lab Code</b>	K9806584-001	<b>Basis</b>	Dry
<b>Test Notes</b>	D		

<b>Analyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>MRL</b>	<b>Dilution Factor</b>	<b>Date Extracted</b>	<b>Date Analyzed</b>	<b>Result</b>	<b>Result Notes</b>
Tetra-n-butyltin	Method	Krone	15	5	11/13/98	11/17/98	ND	UI
Tri-n-butyltin	Method	Krone	5	5	11/13/98	11/17/98	60	
Di-n-butyltin	Method	Krone	5	5	11/13/98	11/17/98	15	
n-Butyltin	Method	Krone	5	5	11/13/98	11/17/98	ND	UI

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

11-25-98

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**COLUMBIA ANALYTICAL SERVICES, INC.**

**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment


**Service Request:** K9806584  
**Date Collected:** 9/22/98  
**Date Received:** 9/23/98

**Butyltins**

**Sample Name:** 98394021 **Units:** ug/Kg (ppb)  
**Lab Code:** K9806584-002 **Basis:** Dry  
**Test Notes:** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	15	5	11/13/98	11/18/98	ND	UI
Tri-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	23	
Di-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	15	
n-Butyltin	Method	Krone	5	5	11/13/98	11/18/98	ND	UI

D The MRL is elevated because of matrix interferences and because the sample required diluting

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*11/23/99*



## Analytical Report

**Service Request:** K9806584  
**Date Collected:** 9/22/98  
**Date Received:** 9/23/98

## Butyltins

Units	ug/Kg (ppb)
Basis	Dry

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result Notes
	Method	Method		Factor	Extracted	Analyzed		
Tetra-n-butyltin	Method	Krone	15	5	11/13/98	11/18/98	ND	UI
Tri-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	13	
Di-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	7	
n-Butyltin	Method	Krone	5	5	11/13/98	11/18/98	ND	UI

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/23/98  
**Date Received:** 9/24/98

## Butyltins

**Sample Name** 98394027  
**Lab Code** K9806584-013  
**Test Notes**

**Units** ug/Kg (ppb)  
**Basis** Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	3	1	11/13/98	11/18/98	4	
Tri-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	3	
Di-n-butyltin	Method	Krone	2	1	11/13/98	11/18/98	ND	B
n-Butyltin	Method	Krone	18	1	11/13/98	11/18/98	ND	UIJ B

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**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/23/98  
**Date Received:** 9/24/98

**Butyltins**

**Sample Name** 98394028 **Units** ug/Kg (ppb)  
**Lab Code** K9806584-014 **Basis** Dry  
**Test Notes**

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	3	1	11/13/98	11/18/98	ND	
Tri-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
Di-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
n-Butyltin	Method	Krone	17	1	11/13/98	11/18/98	ND	UIJ B

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/23/98  
**Date Received:** 9/24/98

## Butyltins

**Sample Name** 98394029  
**Lab Code** K9806584-015  
**Test Notes**

**Units** ug/Kg (ppb)  
**Basis** Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	3	1	11/13/98	11/18/98	5	J
Tri-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
Di-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
n-Butyltin	Method	Krone	13	1	11/13/98	11/18/98	ND	W I J ✓

B

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client: Roy F Weston, Inc  
Project: Duwamish River/4000-027-001-2019-38  
Sample Matrix: Sediment

Service Request: K9806584  
Date Collected: 9/23/98  
Date Received: 9/24/98

## Butyltins

Sample Name 98394030 Units ug/Kg (ppb)  
Lab Code K9806584-005 Basis Dry  
Test Notes

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	3	1	11/13/98	11/18/98	5 J	
Tri-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
Di-n-butyltin	Method	Krone	1	1	11/13/98	11/18/98	ND	
n-Butyltin	Method	Krone	15	1	11/13/98	11/18/98	ND	W I J ✓

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Date

11-26-98

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/23/98  
**Date Received:** 9/24/98

## Butyltins

**Sample Name** 98394033 **Units** ug/Kg (ppb)  
**Lab Code** K9806584-008 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Krone	15	5	11/13/98	11/18/98	ND	UI
Tri-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	20	
Di-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	8	
n-Butyltin	Method	Krone	5	5	11/13/98	11/18/98	ND	UI

D

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Date

11-25-98

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**Analytical Report**

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9806584  
**Date Collected:** 9/23/98  
**Date Received:** 9/24/98

**Butyltins**

<b>Sample Name</b>	98394034	<b>Units</b>	ug/Kg (ppb)
<b>Lab Code</b>	K9806584-009	<b>Basis</b>	Dry
<b>Test Notes</b>	D		

<b>Analyte</b>	<b>Prep Method</b>	<b>Analysis Method</b>	<b>MRL</b>	<b>Dilution Factor</b>	<b>Date Extracted</b>	<b>Date Analyzed</b>	<b>Result</b>	<b>Result Notes</b>
Tetra-n-butyltin	Method	Krone	15	5	11/13/98	11/18/98	ND	UI
Tri-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	47	
Di-n-butyltin	Method	Krone	5	5	11/13/98	11/18/98	21	
n-Butyltin	Method	Krone	5	5	11/13/98	11/18/98	7	J

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